IS 8877 (1987): Specification for Acetoacetic Methyl Ester [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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(Reaffirmed 2008)

Indian Standard

SPECIFICATION FOR ACETOACETIC METHYL ESTER

(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

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Indian Standard

SPECIFICATION FOR ACETOACETIC METHYL ESTER

(First Revision)

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Indian Standard

SPECIFICATION FOR ACETOACETIC METHYL ESTER

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 2 February 1987, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Acetoacetic methyl ester ($C_5H_8O_3$) is an important organic intermediate used in the manufacture of dyestuffs. It is represented by the following chemical formula:

$CH_3.CO.CH_2.CO.OCH_3$
ACETOACETIC METHYL ESTER
(Molecular Mass 116)

0.3 This standard was first published in 1978. The Committee responsible for the preparation of this standard decided to revise it in order to update the requirements and also to introduce the gas chromatographic method for assay.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for acetoacetic methyl ester.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless liquid.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR ACETOACETIC METHYL ESTER

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	98·5	A-1
ii)	Acidity (as CH_3COOH), percent by mass, *Max*	0·2	A-2
iii)	Moisture, percent by mass, *Max*	0·1	A-3

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979*) or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net and gross mass.

*Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Each individual sample shall be tested for assay.

4.2.2 Tests for remaining characteristics, namely, acidity and moisture shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR ACETOACETIC METHYL ESTER

A-1. ASSAY

A-1.0 General — Two methods have been prescribed for assay: (a) hydroxylamine hydrochloride method and (b) gas chromatographic method. In case of dispute, gas chromatographic method shall be used as the referee method.

A-1.1 Hydroxylamine Hydrochloride Method

A-1.1.0 *Outline of the Method* — Acetoacetic methyl ester reacts with hydroxylamine hydrochloride and liberates hydrochloric acid which is estimated.

A-1.1.1 *Reagents*

A-1.1.1.1 *Methanol*

A-1.1.1.2 *Hydroxylamine hydrochloride solution* — approximately 1 N. Prepare as follows:

Weigh about 35 g of pure hydroxylamine hydrochloride in a beaker, dissolve it in about 100 ml of water and make it up to 500 ml in a standard volumetric flask with water.

A-1.1.1.3 *Sodium hydroxide solution* — 1 N.

A-1.1.1.4 *Methyl orange indicator*

A-1.1.1.5 *Methylene blue indicator*

A-1.1.2 *Procedure* — Weigh accurately about 2·5 to 3 g of sample in a 250-ml stoppered conical flask. Add to this 50 ml of methanol followed by 50 ml hydroxylamine hydrochloride. Mix well and allow to stand for one to one and a half hours at room temperature with occasional shaking. Add 5 drops of methyl orange indicator and 5 drops of methylene blue indicator and titrate against sodium hydroxide solution till colour change is from violet to light green. Take a blank in the similar way.

A-1.1.3 *Calculation*

$$\text{Assay, percent by mass} = \frac{(V_1 - V_2) \times 11{\cdot}6 \times N}{M}$$

where

V_1 = volume in ml of sodium hydroxide solution required for the sample,

V_2 = volume in ml of the sodium hydroxide solution required for the blank,

N = normality of sodium hydroxide solution, and

M = mass in g of sample taken for the test.

A-1.2 Gas Chromatographic Method

A-1.2.0 *Outline of the Method* — The assay of acetoacetic methyl ester is determined by gas liquid chromatographic method. A sample of the material is injected into the gas chromatograph apparatus where it is carried by the carrier gas from one end of the column to the other end. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after the other and are detected by suitable means where response is related to the amount of a specific component leaving the column.

A-1.2.1 *Apparatus* — Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram obtained with Perkin Elmer Σ 3B instrument and the following conditions are shown in Fig. 1:

Column	:	Material — Stainless steel
		Dimension — Length 2 000 mm; internal diameter, 2·75 mm
		Stationary phase — 10 percent XE 60 on Chromosorb WAW/DMCS, mesh 80/100
Carrier gas	:	Nitrogen
Flow rate	:	30 ml/minute
Injector temperature	:	200°C
Detector temperature	:	200°C
Condition	:	Oven temperature

a) initial temperature, 50°C for 3 minutes, and

b) final temperature, 100°C with a programming rate of 30°C/minute

Detector	:	Frame ionization
Chart speed	:	5 mm/minute
Sample size	:	0·5 microlitre
Retention time	:	acetoacetic methyl ester, 8·71 minutes

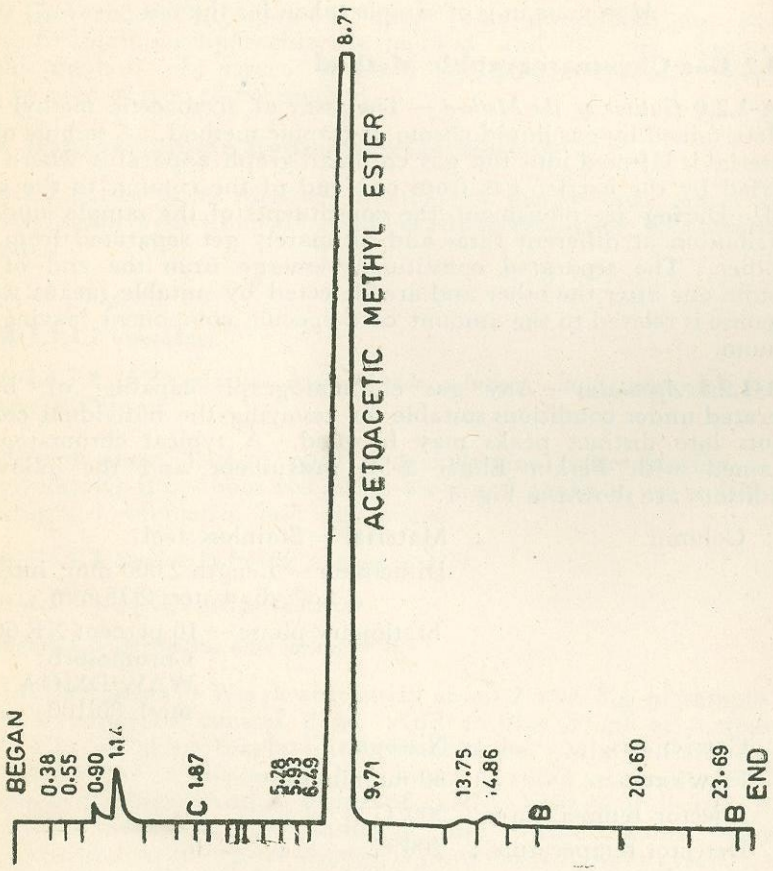

FIG. 1 TYPICAL CHROMATOGRAM

A-1.2.2 *Procedure* — Conduct the flow of the carrier gas and inject 0·5 microlitre of the sample at injection port where it is vapourized and well mixed with carrier gas. This is led into the chromatographic

column wherein the vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For an efficient separation, the column should be maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives a signal corresponding to the amount of particular constituent leaving the column. The detector signal on transmission to the recorder plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-1.2.3 *Calculation* — Calculate the peak areas of individual constituent pertaining to acetoacetic methyl ester as also the other constituents and calculate the purity of the sample as given below:

A_1 = area under acetoacetic methyl ester peak;

A_2 = area under other peaks, say peak 1; and

A_3 = area under other peaks, say peak 2, etc.

$$\text{Assay, percent} = \frac{A_1}{A_1 + A_2 + A_3 + \ldots\ldots\ldots + A_n} \times 100$$

where

n = number of other peaks observed apart from acetoacetic methyl ester.

A-2. DETERMINATION OF ACIDITY

A-2.1 Reagents

A-2.1.1 *Mixed Indicator* — Dissolve 0·05 g of bromocresol green and 0·1 g of methyl red in ethanol and make up to 100 ml.

A-2.1.2 *Sodium Hydroxide Solution* — 0·1 N.

A-2.2 Procedure — Weigh accurately about 5 to 6 g of the material in a 250-ml conical flask and add about 100 ml of water and shake it till a good dissolution is obtained. Add a few drops of mixed indicator till the colour changes to red. Titrate the solution with sodium hydroxide solution till colour changes from red to green.

A-2.3 Calculation

$$\text{Acidity, percent by mass} = \frac{V \times 0{\cdot}6}{M}$$

where

V = volume in ml of sodium hydroxide used for titration, and

M = mass in g of the sample taken.

A-3. DETERMINATION OF MOISTURE

A-3.1 Determine the moisture content of the material as prescribed in **9** of IS : 5299-1969*.

*Methods of sampling and tests for dye intermediates.

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